

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 08-030017

(43)Date of publication of application : 02.02.1996

(51)Int.Cl.

G03G 9/08
G03G 9/087
G03G 9/097

(21)Application number : 06-180493

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(22)Date of filing : 11.07.1994

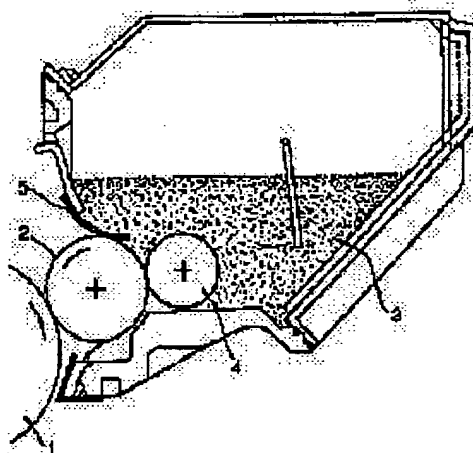
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(54) ELECTROSTATIC CHARGE IMAGE DEVELOPING ONE-COMPONENT TONER

(57)Abstract:

PURPOSE: To provide an electrostatic charge image developing one-component toner having excellent durability without causing contamination of a sleeve, cleaning defects or electrification defects so that good images can be maintained.

CONSTITUTION: This electrostatic charge image developing one-component toner contains a resin charge controlling agent (I) and is used for a developing device in which an electrifying member 5 is pressed to touch a developer carrier body 2 arranged to face a latent image holding body 1. The toner consists of a copolymer having 2000 to 15000 weight average mol.wt. and components of (a) 2- acrylamide-2-methylpropane sulfonic acid, (b) polymerizable monomers having carboxyl groups, (c) styrene and/or α -methylstyrene, and (d) polymerizable monomers having hydroxyl groups. Proportions of these components are specified that (a):(b) = 95:5 to 70:30, (a) and (b):(c) = 3:97 to 20:80, (a) and (b) and (c):(d)= 100 pts.wt.:(0.05 to 2.0 pts.wt.), and (a) and (b):(d):100 pts.wt.: (20 pts.wt. or less).



LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

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CLAIMS

[Claim(s)]

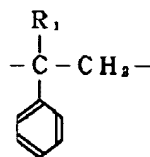
[Claim 1] The 1 component toner for electrostatic-charge image development used for the developer by which the pressure welding of the electrification grant member is carried out to the developer support containing the binding resin and the resin charge control agent which countered the latent-image supporter and has been arranged.

[Claim 2] The 1 component toner for electrostatic-charge image development according to claim 1 characterized by this resin charge control agent containing following (I) or the resin charge control agent of (II).

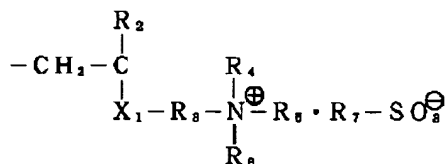
Resin charge control agent (I)

Following (a), (b), (c) And each component composition of (d) The copolymer of the weight average molecular weight 2,000-15,000 which it has alpha-methyl-styrene [2-acrylamide-isobutane sulfonic-acid (b) carboxyl group content polymerization nature monomer (c) styrene and/or] (d) hydroxyl content polymerization nature monomer component (a):component (b) = component (a) (a) -- 95:5 - 70:30% And the (Component c):(component d) = 100 section: 0.05 - 2.0 section resin charge control agent (II) [a (Component b):(component c) = 3:97-20:80% component (a), a component (b), and] The weight average molecular weight which has each repeat unit of following (e), (f), and (g) is the copolymer of 2,000-20,000. [Formula 1]

(e)

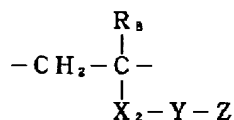
(R₁は水素原子、炭素数1～3のアルキル基)

(f)



(R₂は水素原子、メチル基、R₃はアルキレン基、
 R₄, R₅, R₆は炭素数1～6のアルキル基、
 R₇はメチル基、エチル基、プロピル基、フェニル基、メチルフェニル基、
 ナフチル基、X₁は-COO-, -CONH-である。)

(g)



(R₉は水素原子、メチル基、X₂は-COO-, -CONH-)
 (Yはアルキレン基、Zは水素原子、水酸基である。)

The repeat unit = 100 weight section of a component (repeat unit: component (g) of 65 - 97 % of the weight of repeat units of e), 35 - 3 % of the weight of repeat units of a component (f), a component (e), and a component (f)): 0.1 - 2.0 weight section. [Claim 3] The 1 component toner for electrostatic-charge image development whose each melt viscosity η_{PB} and η_{PC} in each softening temperature the temperature gradient of the softening temperature (SPB) of the binding resin measured by the flow tester and the softening temperature (SPC) of a resin charge control agent according to claim 2 is 30 degrees C or less, and are $5 \times 10^{-2} \leq \eta_{PC} / \eta_{PB} \leq 10$.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the 1 component toner used in order to visualize notably the electrostatic-charge latent image in electrophotography and electrostatic recording.

[0002]

[Description of the Prior Art] Various methods are indicated by a U.S. Pat. No. 2,297,691 specification, JP,42-23910,B (U.S. Pat. No. 3,666,363 specification), JP,43-24748,B (U.S. Pat. No. 4,071,361 specification), etc. as a conventional xerography. The development method applied to these xerographies is divided roughly into the dry-developing method and the wet-developing method, and the former is further divided into the method of using an one-component-system developer, and the method using a binary system developer.

[0003] The coloring agent with which a toner consists of a color, a pigment, etc. into nature or a composite binding resin, a charge control agent, the magnetic substance, a wax, etc. are inner-**(ed), and the particle pulverized to about 1-30 micrometers is used. Furthermore, plasticizers, such as a silica, may be mixed. In order to be charged in positive or negative and to make a toner hold a charge according to the polarity of the electrostatic-charge latent image by which any toner is developed, adding a charge control agent is performed.

[0004] The triboelectrification of a toner is urged to a charge control agent, it controls the amount of triboelectrification, and carries out the work which maintains the amount of triboelectrification. As a typical charge control agent of negative electrification nature, the metal complex compound of a carboxylic acid, a diazo compound, the complex compound by boron, etc. are mentioned, and colors and pigments, such as a quarternary-ammonium-salt compound, an imidazole compound, a Nigrosine, an azine system color, and a triphenylmethane-color system color, are mentioned as a typical charge control agent of right electrification nature. Since compatibility with a binding resin is bad compared with the charge control agent of a resin, what exists in a toner front face tends to break away, and these charge control agents have the fault which is easy to cause the variation in electrification of a toner, and sleeve contamination. Then, as a charge control agent which can improve, in JP,63-184762,A and JP,3-56974,A, a negative electrification nature resin charge control agent is indicated, and the right electrification nature resin charge control agent is indicated by JP,63-60458,A and JP,3-80259,A. Since compatibility with a binding resin is good, the stable electrification property is acquired, and since transparency is good, use of a color toner is possible for these resin charge control agents also by mixture with a binding resin. However, compared with charge control agents, such as a metal complex compound and an azo dye system, it is a fault that the amount of electrifications is low, it is necessary to use it in a two component developer or, and this charge control agent needs to increase the addition of this charge control agent in a toner.

[0005] If the addition of this charge control agent increases, since we will be anxious about the fall of the compatibility to a binding resin, and generating of offset, improvement in the compatibility to the binding resin of this further charge control agent is desired.

[0006] By the way, although the touchdown developing-negatives method of a U.S. Pat. No. 4,121,931 specification etc. is mentioned as the development method using the one-component-system developer which consists only of a toner, the jumping development method is proposed in JP,55-18656,A etc. Moreover, the 1 component development method shown in JP,58-116559,A, JP,60-120368,A, JP,63-271371,A, etc. carries out the coat of the developer with a blade etc. on a developer support, and since a developer is charged by friction with a blade or a developer support body surface, it can attain improvement in the electrification capacity of a toner compared with a non-contact case. By this method, as for the blade quality of the material, what is easy to be charged in polarity contrary to a developer is desirable, and adding the electrification control component of a toner and reversed polarity in a blade to JP,61-250666,A, JP,62-24285,A, JP,62-203177,A, JP,63-81376,A, JP,63-250662,A, etc. is proposed. By these means, even if electrification is low, an early electrification property improves remarkably. However, the toner received the damage by blade ** etc. by long-term use, and the charge control agent which it becomes easy to exfoliate the additive which exists in a toner front face, and is easy to be charged itself also especially in it had the problem of adhering to a blade or a sleeve and polluting a front face.

[0007]

[Problem(s) to be Solved by the Invention] The purpose of this invention does not have sleeve contamination, poor cleaning, and toner degradation, and even if the developer by which the pressure welding of the electrification grant member is carried out to the developer support which countered the latent-image supporter and has been arranged from the property excellent in endurance is used for it, it is to offer the 1 component toner for electrostatic-charge image development which can continue maintaining a good picture.

[0008]

[Means for Solving the Problem and its Function] As a result of this invention person's inquiring wholeheartedly, the latent-image supporter was countered, and it has been arranged and found out that the above-mentioned trouble was solvable by using the toner which contained the resin charge control agent in the developer by which the pressure welding of the electrification grant member is carried out to the developer support. Use of the high resin charge control agent of safety while the environmental load of a chemical is asked recently is effective. This charge control agent is employing a point excellent in compatibility with a binding resin efficiently, and secession of the charge control agent by the mechanical and thermal load of a toner is suppressed, and it can improve the endurance of a toner. However, although excelled in electrification stability, since the amount of electrifications was insufficient, the toner containing this charge control agent had a problem in ***** or environmental stability. Then, this invention person found out that the amount of electrifications of this toner could be raised with friction with the electrification grant member which is carrying out the pressure welding to a development support and it. moreover, electrification grant -- also according to the kind of member, since the amount of electrifications of this toner was controllable, that a charge up can also be prevented also found out

[0009] Although an example of the developer for nonmagnetic 1 component toners used for this invention is explained, it is not necessarily limited to this. In drawing 1, 1 is a latent-image supporter and latent-image formation is accomplished by the electrophotography process means or electrostatic recording means which is not illustrated. 2 is a developer support and consists of aluminum or a stainless nonmagnetic sleeve. Although the developer support 2 may use aluminum and a stainless rough pipe as it is, what carried out the coat by what sprayed the glass bead etc. and damaged the front face uniformly preferably, the thing which carried out mirror-plane processing, or the resin is good. The developer is stored in the hopper 3 and supplied to up to the developer support 2 by the feed roller 4. the relative velocity which the feed roller 4 consists of foam, such as a polyurethane foam, and is not 0 to the developer support 2 at order or an opposite direction -- having -- rotating -- developer supply -- the developer after the development on the developer support 2 (developer non-developed negatives) -- also stripping off -- it is carrying out the developer supplied on the developer support 2 -- electrification grant -- it is made homogeneity and a thin layer with ** by the elastic blade which is a member 5

electrification grant -- the contact pressure force of a member 5 and the developer support 2 has preferably effective 10 - 120 g/cm three to 250 g/cm as a linear pressure of the direction of a sleeve bus-bar When the contact pressure force is smaller than cm 3g /, it becomes difficult with [of a toner] uniform **, and the amount distribution of electrifications of a toner becomes broadcloth, and causes fogging and scattering. Moreover, if the contact pressure force exceeds 250 g/cm, since a big pressure will be applied to a developer and the external additive of a developer will deteriorate, it is not desirable that condensation of a developer occurs etc. Moreover, it is not desirable in order to require torque big in order to make the developer support 2 drive. electrification grant -- as for a member 5, it is desirable to use the thing of the quality of the material of a triboelectrification sequence made suitable for a developer being charged in desired polarity For example, in order to just electrify a developer, if silicone rubber, polyurethane, a fluororubber, poly chloro butadiene rubber, etc. use styrene butadiene rubber, nylon, etc. as a blade in order to electrify negative, the triboelectrification efficiency of a developer will become higher. moreover -- even blending a silica, a resin particle, etc. -- electrification grant -- the triboelectrification grant nature to the developer of a member 5 can be adjusted moreover, blending electric conduction powder, such as carbon and titanium oxide, etc. -- carrying out -- electrification grant -- it can prevent charging a developer superfluously by giving moderate conductivity to a member 5

[0010] The resin charge control agent (I) which can be used for the toner of this invention is the copolymer of the weight average molecular weight 2,000-15,000 which has each component composition of following (a), (b), (c), and (d).

[0011] 2-acrylamide-isobutane sulfonic-acid (b) carboxyl group content polymerization nature monomer (c) styrene and/or alpha-methyl-styrene (d) hydroxyl content polymerization nature monomer component (a) : (a) (Component b)=95:5 - 70:30% component (a) And the (Component c):(component d)=100 section: The weight average molecular weight of a 0.05 - 2.0 section resin charge control agent (I) needs to be 2,000-15,000. [a (Component b):(component c)=3:97-20:80% component (a), a component (b), and] It is 3,000-8,000 preferably. It is in the inclination for the distribution to a binding resin to become bad if the moisture resistance of a toner falls less than in 2,000, and weight average molecular weight is in the inclination offset becomes easy to generate and exceeds 15,000, and for fogging and a ghost to become bad and is not desirable.

[0012] (b) As a carboxyl group content polymerization nature monomer, an acrylic acid, a methacrylic acid, a maleic acid, a fumaric acid, an itaconic acid, a crotonic acid, etc. are mentioned, for example.

[0013] (d) As a hydroxyl content polymerization nature monomer, hydroxyethyl methacrylate, hydroxyethyl acrylate, hydroxy isopropyl acrylate, etc. are mentioned, for example.

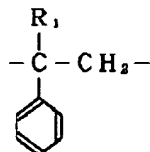
[0014] Moreover, a component (a): It is desirable that a component (b) is 95:5 - 70:30%, and it is more preferably [90:10 - 80:20% of] good. a component (a) -- 95% -- exceeding (a component (b) being less than 5%) -- dispersibility of the magnetic substance in a toner, a coloring agent, and the inner ** agent of a wax and others may be worsened, and a component (a) is [the environmental stability of a toner] in the inclination which becomes bad and is not desirable at less than (a component (b) exceeds 30%) 70%

[0015] A component (a) and a component (b): It is desirable that a component (c) is 3:97 - 20:80%, and it is more preferably [5:95 - 13:87% of] good. a component (a) and a component (b) -- 20% -- exceeding (a component (c) being less than 80%) -- it is [it is in the inclination for the amount of electrifications of a toner to become low under highly humid, and / a component (a) and a component (b)] in the inclination for the electrification ability of a resin charge control agent to become low at less than (for a component (c) to exceed 97%) 3% and is not desirable

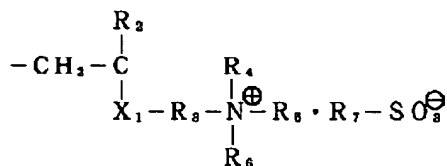
[0016] A component (a), a component (b), and a component (c): It is desirable that a component (d) is the 100 section:0.05 - 2.0 section, and the 100 section:0.1 - 1.5 section is more preferably good. It is in the inclination for the dispersion effect in a toner particle to no longer be obtained and is not desirable if a component (d) exceeds the less than 0.05 sections or the 2.0 sections.

[0017] The weight average molecular weight in which the resin charge control agent (II) which can be used for the toner of this invention has each repeat unit of following (e), (f), and (g) is the copolymer of 2,000-20,000.

[0018]

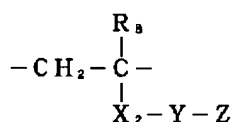
[Formula 2]
(e)(R₁は水素原子、炭素数1～3のアルキル基)

(f)



(R₂は水素原子、メチル基、R₃はアルキレン基、
R₄, R₅, R₆は炭素数1～6のアルキル基、
R₇はメチル基、エチル基、プロピル基、フェニル基、メチルフェニル基、
ナフチル基、X₁は-COO-, -CONH-である。)

(g)



(R₈は水素原子、メチル基、X₂は-COO-, -CONH-
Yはアルキレン基、Zは水素原子、水酸基である。)

[0019] The repeat unit = 100 weight section of a component (repeat unit: component (g) of 65 - 97 % of the weight of repeat units of e), 35 - 3 % of the weight of repeat units of a component (f), a component (e), and a component (f): As for the weight average molecular weight of a 0.1 - 2.0 weight section resin charge control agent (II), it is desirable that it is 2,000-20,000, and 5,000-16,000 are more preferably good. It is in the inclination for distribution with a binding resin to become bad if the moisture resistance of a toner falls less than in 2,000, and weight average molecular weight is in the inclination offset becomes easy to generate and exceeds 20,000, and for fogging and a ghost to become bad and is not desirable.

[0020] Moreover, as for the repeat unit of a component (e), it is desirable that it is 65 - 97 % of the weight, and it is still more preferably good 77 to 97% of the weight more preferably. [of 80 - 95 % of the weight] At less than 65 % of the weight, if it is in the inclination for compatibility with a binding resin to get worse and exceeds 97 % of the weight, although compatibility with a binding resin will become good, it is in the inclination for the environmental stability of a toner to become bad and is not desirable.

[0021] As for the repeat unit of a component (f), it is desirable that it is 35 - 3 % of the weight, and it is still more preferably good 23 to 3% of the weight more preferably. [of 20 - 5 % of the weight] At less than 3 % of the weight, if it is in the inclination for the electrification ability as a charge control agent to fall and exceeds 35 % of the weight, it is in the inclination for moisture resistance to fall and is not desirable.

[0022] Furthermore, the repeat unit of a component (e) and a component (f): It is desirable that the repeat unit of a component (g) is the 100 weight section: 0.1 - 2.0 weight section, and the 100 weight section: 0.3 - 1.5 weight section is more preferably good. It is in the inclination for the distribution in a toner to become bad and is not desirable if the 2.0 weight sections are exceeded under the 0.1 weight section.

[0023] inner **** -- desirable -- the binding resin 100 weight section -- receiving -- 0.1 - 10 weight section -- it is 0.5 - 6 weight section more preferably

[0024] Moreover, when each melt viscosity η_{PB} and η_{PC} in the softening temperature (SPB) of a binding resin and the softening temperature (SPC) of this resin charge control agent which were measured by the flow tester are the relation of $5 \times 10^{-2} \leq \eta_{PC}/\eta_{PB} \leq 10$, in order that this invention persons may raise the amount of electrifications in this toner It found out that the 1 component toner excellent in sleeve contamination, poor cleaning, and the endurance with poor electrification that is not was obtained, without both compatibility falling, even if the addition of this charge control agent increased.

[0025] At this time, it is desirable that the difference of SPB and SPC is smaller than 30 degrees C. Moreover, as for η_{PC}/η_{PB} , it is desirable that it is in the range of 5×10^{-2} to 1. When η_{PC}/η_{PB} is larger than 5×10^{-2} to less than 2, and 10, the compatibility of the inside of a binding resin and this resin charge control agent is bad. When the developer by which the pressure welding of the electrification grant member is carried out to the developer support which the distributed state of this resin charge control agent in a toner became bad, countered the latent-image supporter, and has been arranged is used, Since secession of this resin charge control agent takes place in the portion to which the pressure welding of the electrification grant member is carried out, sleeve contamination, poor cleaning, and poor electrification take place.

[0026] Next, the measuring method of the viscosity in connection with this invention is explained.

[0027] Using the elevated formula flow tester (Shimazu flow-tester CFT-500 type) shown in drawing 2, the viscosimetry of a binding resin or a toner applied the load of 10Kgf(s) by the plunger under constant temperature, extruded about 1.5g sample first fabricated using the pressing machine from the nozzle with a diameter [of 1mm], and a length of 1mm, and, thereby, measured the amount of plunger descent of a flow tester (exit velocity). This exit velocity can be measured at each temperature (it is 5-degree-C interval about a 100 degrees C - 180 degrees C temperature requirement), and it can ask for apparent viscosity η' by the following formula from this value.

[0028]

[Equation 1]

$$\eta' = \frac{TW'}{DW'} = \frac{\pi PR^4}{8 L Q} \quad (\text{p o i s e})$$

但し、

$$TW' = \frac{PR}{2 L} \quad (\text{d y n e / c m}^2)$$

$$DW' = \frac{4 Q}{\pi R^3} \quad (\text{s e c}^{-1})$$

[0029] η' : Apparent viscosity (poise)

TW' : Shearing stress of the appearance of a tube wall (dyne/cm²)

DW' : The shear rate of the appearance of a tube wall (1/sec)

Q : Exit velocity (cm³/sec=ml/sec)

P : Extrusion pressure (dyne/cm²) [10Kgf=980x10⁴dyne]

R : The radius of a nozzle (cm)

[0030] The softening temperature SP here is the intermediate temperature of the temperature T1 into which the whole sample is melted and stabilized and begins to flow with a temperature up, and the temperature T2 with which exit velocity was saturated in flow-tester measurement.

[0031] The toner of this invention adds a resin charge control agent, a coloring agent, the magnetic substance, a wax, etc. to a binding resin. The amount of these compounds used is not determined by the toner manufacture method including the kind of binding resin, the existence of the additive used if needed, and the distributed method, and is not limited uniquely.

[0032] As a resin for forming the toner particle used for this invention For example, styrene, such as polystyrene, Poly p-crawl styrene, and polyvinyl toluene, and the single polymer of the substitution product; A styrene-p-crawl styrene copolymer, A styrene-vinyltoluene copolymer, a styrene-vinyl naphthalene copolymer, A styrene-acrylic-ester copolymer, a styrene-methacrylic-ester copolymer, A styrene-alpha-crawl methyl-methacrylate copolymer, a styrene acrylonitrile copolymer, A styrene-vinyl methyl-ether copolymer, a styrene-vinyl ethyl ether copolymer, A styrene-vinyl methyl ketone copolymer, a styrene-butadiene copolymer, Styrene system copolymers, such as a styrene-isoprene copolymer and a styrene-acrylonitrile-indene copolymer; A polyvinyl chloride, Phenol resin, natural denaturation phenol resin, natural resin denaturation maleic resin, Acrylic resin, methacrylic resin, polyvinyl acetate, silicone resin, polyester resin, polyurethane, polyamide resin, a furan resin, an epoxy resin, a xylene resin, a polyvinyl butyral, a terpene resin, a cumarone indene resin, a petroleum system resin, etc. can be used.

[0033] Moreover, the styrene system copolymer over which the bridge was constructed is also a desirable binding resin. As a comonomer to the styrene monomer of a styrene system copolymer For example, an acrylic acid, a methyl acrylate, an ethyl acrylate, a butyl acrylate, An acrylic-acid dodecyl, an acrylic-acid octyl, acrylic-acid-2-ethylhexyl, An acrylic-acid phenyl, a methacrylic acid, a methyl methacrylate, an ethyl methacrylate, The monocarboxylic acid which has appearance double bonds, such as methacrylic-acid butyl, a methacrylic-acid octyl, acrylonitrile, a methacrylonitrile, and an acrylamide, or its substitution product; for example The dicarboxylic acid which has double bonds, such as a maleic acid, maleic-acid butyl, a maleic-acid methyl, and a maleic-acid dimethyl, and substitution-product;, for example, a vinyl chloride, Vinyl-esters;, for example, ethylene, such as vinyl acetate and a benzoic-acid vinyl, Ethylene system olefins;, for example, vinyl methyl ketones, such as a propylene and a butylene, vinyl monomers, such as vinyl ether [, such as vinyl ketones /, such as a vinyl hexyl ketone, /;, for example a vinyl methyl ether, vinyl ethyl ether, and the vinyl isobutyl ether,],, are independent -- or two or more are used The carboxylate which the derivative which mainly has the double bond in which two or more polymerizations are possible as a cross linking agent here is used, for example, has two double bonds, such as aromatic divinyl compound [, such as a divinylbenzene and divinyl naphthalene,],, for example, ethylene glycol diacrylate, ethylene glycol dimethacrylate, 1, and 3-butanediol dimethacrylate;, derivative; which has divinyl derivative [, such as a divinyl aniline, the divinyl ether, a divinyl sulfide, and a divinyl sulfone,]; and three or more vinyl groups is used as independent or mixture.

[0034] moreover -- the case where a pressurization fixing method is used -- use of the binding resin for pressure fixing toners -- possible -- for example, polyethylene, polypropylene, a polymethylene, a polyurethane elastomer, an ethylene-ethyl acrylate copolymer, an ethylene vinylacetate copolymer, an ionomer resin, a styrene-butadiene copolymer, a styrene-isoprene copolymer, and a line -- there are a saturated polyester, paraffin, etc.

[0035] It is a desirable gestalt to ** a silica outside to the toner containing this resin charge control agent of this invention.

[0036] although the amount of the silica used in that case is not what is limited uniquely -- desirable -- the toner 100 weight section -- receiving -- 0.05 - 10 weight section -- it is more preferably used in the range of 0.2 - 5 weight section

[0037] As such a silica impalpable powder, the silica impalpable powder manufactured with dry process and the wet method can be used. The dry process said here is the manufacture method of the silica pulverized coal generated by steamy phase oxidization of a silicon halogen derivative, and it is also possible by using other metal halogen derivatives, such as an aluminum chloride or a titanium chloride, with a silicon halogen derivative in a manufacturing process to obtain the compound pulverized coal of a silica and other metallic oxides. The method of on the other hand manufacturing a silica pulverized coal with a wet method can apply conventionally well-known various methods. For example, after making an alkaline-earth-metal silicate generate from decomposition by the ammonia salts or the alkali salts of decomposition by the acid of a sodium silicate, and a sodium silicate, and a sodium silicate, there is a method of using the method of decomposing from an acid and making it into a silicic acid, the method

of making a sodium-silicate solution a silicic acid by ion exchange resin, a natural silicic acid, or a silicate etc.

[0038] Each silicate, such as an anhydrous silicon dioxide (silica), other aluminum silicates, a sodium silicate, a potassium silicate, a magnesium silicate, and silicic-acid zinc, is applicable to a silica pulverized coal here.

[0039] A result with a specific surface area good [the thing within the limits more than 30m²/g (especially 50-400m²/g)] among the above-mentioned silica pulverized coals by the nitrogen adsorption measured by the BET adsorption method is given.

[0040] Moreover, the silica pulverized coal used for this invention may be processed by processing agents, such as a silane coupling agent and an organic silicon compound, if needed, and the method that the method is also well-known is used, and it is processed by the silica pulverized coal, the reaction, or physical adsorption.

[0041] As an example of the processing agent which obtains a right electrification nature silica ** aminopropyl trimethoxysilane, aminopropyl triethoxysilane, dimethylamino propyltrimethoxysilane, diethylamino propyltrimethoxysilane, dipropylamino propyltrimethoxysilane, dibutylamino propyltrimethoxysilane, Monobutyl aminopropyl trimethoxysilane, dioctylamino propyltrimethoxysilane, dibutylamino propyl dimethoxysilane, a dibutylamino PUROPIRUMONO methoxy silane, dimethylamino phenyl triethoxysilane, There are trimethoxysilyl-gamma-propyl phenylamine, trimethoxysilyl-gamma-propyl benzylamino, etc. by the nitrogen-containing heterocycle system There are a trimethoxysilyl-gamma-propyl piperidine, a trimethoxysilyl-gamma-propyl morpholine, a trimethoxysilyl-gamma-propyl imidazole, etc.

[0042] moreover, as an example of the processing agent which obtains a negative electrification nature silica A hexamethyldisilazane, a ** RIMECHIRU silane, a trimethyl crawl silane, A trimethyl ethoxy silane, a dimethyl dichloro silane, methyltrichlorosilan, An allyl-compound dimethyl crawl silane, an allyl-compound phenyl dichloro silane, a benzyl dimethyl crawl silane, Bromine methyl dimethyl KURORUSHIRAN, alpha-crawl ethyl trichlorosilan, beta-crawl ethyl trichlorosilan, KURORUMECHIRU dimethyl KURORUSHIRAN, A trio luganot silyl mercaptan, a trimethylsilyl mercaptan, Trio luganot silylacrylate, vinyl dimethyl acetoxysilane, A dimethylethoxy silane, dimethyl dimethoxysilane, diphenyl diethoxysilane, Hexa methyl disiloxane, 1, 3-divinyl tetramethyl disiloxane, The dimethylpolysiloxane containing the hydroxyl group combined with Si of one-piece **, respectively etc. is in 1, 3-diphenyl tetramethyl disiloxane, and the unit that has 12 siloxane units from per [2] molecule, and is located in an end. These are used with one sort or two sorts or more of mixture.

[0043] Moreover, as a silicone oil, methyl-silicone oil, a dimethyl silicone oil, a phenylmethyl silicone oil, a crawl phenylmethyl silicone oil, an alkyl denaturation silicone oil, a fatty-acid denaturation silicone oil, polyoxyalkylene denaturation oil, etc. are desirable. These are used with one sort or two sorts or more of mixture.

[0044] In addition, when a right triboelectrification nature silica pulverized coal is measured by the blowing-off method in this invention, what has positive charge to an iron powder carrier is said.

[0045] to a developer weight, the dosage of these processed silica pulverized coals demonstrates an effect at the time of 0.01 - 20%, and when it adds 0.03 to 5% preferably especially, it shows the positive triboelectrification nature which has the stability which was alike and excellent If a desirable mode is described about an addition gestalt, it is good that it is in the state where 0.01 - 3% of the weight of the processed silica pulverized coal has adhered to the toner particle front face to a developer weight.

[0046] The toner for electrostatic-charge image development of this invention makes a magnetic material contain, and can also be used as a magnetic toner. An alloy, its mixture, etc. with a metal like metals, such as iron-oxide; iron, such as a magnetite, a gamma ferric oxide, a ferrite, and an iron-overload type ferrite, cobalt, and nickel, or these metals, aluminum and cobalt, copper, lead, magnesium, tin, zinc, antimony, beryllium, a bismuth, cadmium, calcium, manganese, a selenium, titanium, a tungsten, and vanadium as a magnetic material used are mentioned. as the amount which these magnetic materials have an about 0.1-0.5-micrometer preferably desirable thing 0.1-1 micrometer, and a mean particle diameter makes contain in a magnetic toner -- the binding resinous principle 100 weight section

-- receiving -- the 20 - 150 weight section -- it is the 30 - 120 weight section preferably
 [0047] In the magnetic toner using the charge control agent of this invention, the toner whose weighted-mean particle size is 3-15 micrometers is usable. The magnetic toner particle to which the ***** NA particle to which the magnetic toner particle which has especially the particle size of 5 micrometers or less has 12-60-piece the particle size which it contains several% and is 8-12.7 micrometers contains several 1-33%, and has the particle size of 16 micrometers or more contains 2.0 or less % of the weight, and it is more desirable than the upper shell of a development property that the weighted-mean particle size of a magnetic toner is 4-10 micrometers.

[0048] Although the particle size distribution of a toner can be measured by various methods, it is appropriate to carry out using a Coulter counter in this invention.

[0049] That is, the interface (product made from the department machine of a day) and CX-1 personal computer (Canon make) which output a number distribution and a volume-integral cloth are connected using a Coulter-counter TA-II type (coal tar company make) as a measuring device, and the electrolytic solution adjusts NaCl solution about 1% using the 1st class sodium chloride. For example, ISOTON(R)-II (made in coal tar scientific Japan) can be used. as a measuring method -- the inside of the 100-150ml of the aforementioned electrolysis solution -- as a dispersant -- a surfactant -- 0.1-5ml of alkylbenzene sulfonates is added preferably, and 2-20mg of measurement samples is added further The electrolytic solution which suspended the sample performed distributed processing for about 1 - 3 minutes with the ultrasonic distribution vessel, and it computed the volume-integral cloth of a 2-40-micrometer particle, and the number distribution by having measured the volume of a toner, and the number with the aforementioned Coulter-counter TA-II type, using 100-micrometer aperture as aperture. And it asked for the weight mean diameter (D4) (let the median of each channel be the central value for every channel) of the weight criteria searched for from the volume-integral cloth concerning this invention, the amount of coarse powders of the weight criteria searched for from the volume-integral cloth (20.2 micrometers or more), and the fines number (6.35 micrometers or less) of the number criteria searched for from the number distribution.

[0050] Moreover, the toner of this invention may mix an additive if needed. As an additive, there are conductive grant agents, such as an abrasive material like the lubricant like a zinc stearate or a cerium oxide, and silicon carbide or a fluid grant agent like an aluminum oxide or titanium oxide, a caking inhibitor or carbon black, and tin oxide, for example.

[0051] Moreover, fluorine content polymer impalpable powders, such as a poly vinylidene fluoride impalpable powder, are also desirable additives from points, such as a fluidity, polish nature, and electrification stability.

[0052] Moreover, it is also one of the desirable gestalten of this invention to add wax-like matter, such as low molecular weight polyethylene, low molecular weight polypropylene, a micro crystalline wax, carnauba wax, a SAZORU wax, and paraffin wax, to a toner about 0.5 to 5% of the weight in order to improve the mold-release characteristic at the time of hot-calender-roll fixing.

[0053] After mixing enough a toner component which was mentioned above with the mixer of a ball mill and others in manufacturing the toner concerning this invention, the heat kneading machine of a hot-calender-roll kneader and an extruder may be used, it kneads, and the method of obtaining a toner by mechanical trituration and the classification is desirable after cooling solidification. After distributing a component in a binding resin solution, in the so-called microcapsule toner which consists of method; which obtains a toner or core material, and shell material, methods, such as method; which makes core material, shell material, or these both contain a predetermined material, are applicable to others by carrying out spray drying.

[0054] Furthermore, a desired additive can fully be mixed with mixers, such as a Henschel mixer, if needed, and the toner concerning this invention can be manufactured.

[0055] Conventionally, the toner of this invention is a well-known means, and is altogether usable in the development for developing the electrostatic-charge image in electrophotography, electrostatic recording, electrostatic printing, etc.

[0056]

[Example] The example of manufacture of the resin charge control agent used for below by this invention is shown.

[0057] The agitator, the capacitor, the thermometer, and the gas introduction pipe were attached to the flask of 12l. of examples of manufacture of a resin charge control agent (I), xylene 400g, 24g [of 2-acrylamide-isobutane sulfonic acids], 6g [of methacrylic acids], and styrene 575g, hydroxypropyl methacrylate 1.2g, and benzoyl peroxide 1.5g were put in into the flask, and solution polymerization was carried out under N₂ gas for 90-degree-C 7 hours. The solvent was removed under 120-degree-C reduced pressure after that, jet mill trituration was performed, and the resin charge control agent (I-1) of softening temperature SPC=125 degree C and melt viscosity $\eta_{PC}=5 \times 10^3$ Pa-s at that time was obtained.

[0058] The agitator, the capacitor, the thermometer, and the gas introduction pipe were attached to the flask of 22l. of examples of manufacture of a resin charge control agent (II), xylene 280g, styrene 540g, hydroxy methacrylate 6g, diethylamino ethyl methacrylate 60g, and benzoyl peroxide 10g were put in into the flask, and solution polymerization was carried out under N₂ gas introduction for 90-degree-C 7 hours. After adding methyl sulfonic-acid methyl 73g and xylene 320g after that and making 90 degrees C react for 4 hours, the solvent was removed under 120-degree-C reduced pressure, jet mill trituration was performed, and the resin charge control agent (II-1) of melt viscosity $\eta_{PC}=6 \times 10^3$ Pa-s was obtained in softening temperature SPC=115 degree C and its softening temperature.

[0059] It is made to be the same as that of the example 2 of manufacture of the example of manufacture 3 resin charge control agent (II) of a resin charge control agent (II). Xylene 300g, styrene 528g, 72g [of butyl acrylates], and diethylamino ethyl methacrylate 9g, Put in benzoyl peroxide 8g and solution polymerization is carried out under N₂ gas introduction for 90-degree-C 6.5 hours. Add 12g of Para-toluenesulfonic acid, and xylene 300g after that, make 90 degrees C react for 4 hours, remove a solvent under 120-degree-C reduced pressure, perform jet mill trituration, and it sets to softening temperature SPC=120 degree C and its softening temperature. The resin charge control agent (II-2) of melt viscosity $\eta_{PC}=3.7 \times 10^4$ Pa-s was obtained.

[0060] Example 1 Styrene-butyl acrylate copolymer The 100 sections (SPB=115 degree C, $\eta_{PB}=3.7 \times 10^4$ Pa-s)

Magnetic oxide of iron The 90 sections Low-molecular-weight propylene The three sections Resin control agent (I-1) The five sections [0061] The above-mentioned mixture was kneaded with the biaxial kneading extruder heated by 140 degrees C. Coarse grinding of the cooled kneading object was carried out with the hammer mill, classification removal of superfines and the coarse powder was simultaneously carried out strictly by the hyperfractionation classifier (elbow jet classifier by Nittetsu Mining Co., Ltd.) which pulverized the coarse-grinding object using the pulverizer using the jet stream, and used the Coanda effect for the obtained pulverizing powder, and the negative electrification nature magnetism toner with a weighted-mean particle size (D₄) of 8.0 micrometers was obtained.

[0062] In this toner 100 weight section, the hydrophobic silica (BET specific surface area 160m²/g) 0.6 weight section was mixed by the Henschel mixer, and the 1 component magnetism toner was obtained in it.

[0063] About the obtained magnetic toner, it is the commercial laser beam printer LASER. SHOT When tested by ****(ing) by A404 (urethane blade use) (Canon, Inc. make), from the first stage, the concentration of 1.43 was obtained and, as for picture concentration, after the copy of five omasums was not accepted by 1.40, as for the concentration fall. The amount of electrifications of the developer on the sleeve of the 500th sheet was -8.3 mC/kg. Moreover, sleeve dirt and poor cleaning were not generated.

[0064] Example 2 Styrene-acrylate copolymer The 100 sections (SPB=125 degree C, $\eta_{PB}=9.8 \times 10^3$ Pa-s)

Carbon black The four sections Low molecular weight polypropylene The four sections Resin charge control agent (II-1) The four sections [0065] The right electrification nature 1 component toner with a weighted-mean particle size of 11.3 micrometers was obtained like the example 1 using the above-mentioned material.

[0066] In this toner 100 weight section, the right electrification nature silica 0.6 weight section was

mixed by the Henschel mixer, and the 1 component nonmagnetic toner was obtained in it.

[0067] It converted into what showed the developer of the commercial copying machine NP1215 (Canon, Inc. make) to drawing 1, what changed the elastic blade 5 into the elastic blade which cast silicone rubber with a thickness of 1.2mm in the shape of a sheet was used, and the above-mentioned developer performed the copy test. The concentration of 1.27 was obtained from the first stage, the concentration of 1.30 was obtained by the 100th sheet, and the standup of picture concentration was also good. Moreover, the amount of electrifications of the developer on the sleeve of the 100th sheet was 28 mC(s)/kg. Then, when ***** is continued to 10,000 sheets, there is also no concentration fall, and sleeve dirt was not accepted, either.

[0068] Example 3 Styrene-butyl acrylate copolymer The 100 sections (SPB=125 degree C, etaPB=6.7x104 Pa-s)

Carbon black The four sections Low molecular weight polypropylene The three sections Resin charge control agent (II-1) The five sections [0069] The right electrification nature 1 component toner with a weighted-mean particle size of 10.8 micrometers was obtained for the above-mentioned material like the example 1.

[0070] In this toner 100 weight section, the right electrification nature silica 0.6 weight section was mixed by the Henschel mixer, and the 1 component nonmagnetic toner was obtained in it.

[0071] The copy test was performed like the example 2. The picture concentration of 1.23 was obtained from the first stage, the standup was also good and, as for concentration, the good picture of 1.24 was acquired also for after ***** of eight omasums. The amount of electrifications at this time was 22 mC (s)/kg.

[0072] Example 4 Styrene-methacrylate copolymer The 100 sections (SPB=115 degree C, etaPB=2.7x103 Pa-s)

Magnetic oxide of iron The 80 sections Resin charge control agent (II-2) The four sections Low-molecular-weight propylene The three sections [0073] The right electrification nature magnetism toner with a weighted-mean particle size of 9.6 micrometers was obtained for the above-mentioned material like the example 1. And in this toner 100 weight section, the right electrification nature silica 0.6 weight section was mixed by the Henschel mixer, and, finally the 1 component magnetism toner was obtained in it.

[0074] When the reconstruction machine which changed the commercial blade of family KOPIA PC-10 (Canon, Inc. make) into the silicone rubber blade with this toner performed the copy test, the picture concentration of 1.40 was obtained from the first stage, and after 3 omasum copy was stable with 1.30. The amount of electrifications on the sleeve of a toner was stable before and behind 9 mC(s)/kg.

[0075] The 8.3-micrometer 1 component magnetism toner was obtained for salicylic acid derivatives like 3 ***** and the example 1 instead of the example of comparison 1 resin charge control agent (I-1). When tested by *****(ing) with the laser beam printer used in the example 1, picture concentration fell from per 500th sheet, it became 1.00 or less, and fogging also became bad.

[0076] In manufacture of an example of comparison 2 resin charge control agent (II-1), styrene and diethylamino ethyl methacrylate were set to 590g and 10g, respectively, and solution polymerization was performed similarly. then, the bottom of 120-degree-C reduced pressure after reacting the methyl sulfonic-acid methyl by 14g and making a xylene react at 330g, in addition 90 degrees C for 4 hours -- a solvent -- removing -- jet mill trituration -- carrying out -- softening temperature SPC= -- 120 degrees C (II-3) of resin charge control agents of solution viscosity etaPC=7x103 Pa-s in the softening temperature were obtained

[0077] In the example 2, the resin charge control agent (II-3) 4 section was put in instead of the resin charge control agent (II-1), and the 11.6-micrometer 1 component nonmagnetic toner was obtained.

[0078] When tested by *****(ing) like an example 2, the standup of early concentration was with 1.17, at least the 1,000th sheet of 1.20 and concentration was low, and its fogging of the sheet was also bad at the rigour convex. The amount of electrifications was as low as 8 mC(s)/kg.

[0079] The resin charge control agent (II-4) of melt viscosity etaPC=3.8x105 Pa-s in softening temperature SPC=125 degree C and its softening temperature was obtained using the same compound as

the example 3 of manufacture of an example of comparison 3 resin charge control agent (II-2).

[0080]

Polyester resin The 100 sections (SPB=85 degree C, $\eta_{PB}=2 \times 10^4$ Pa-s)

Magnetic oxide of iron The 80 sections Resin charge control agent (II-4) The five sections Low-molecular-weight propylene The three sections [0081] The 1 component magnetism toner with a weighted-mean particle size of 12.1 micrometers was obtained for the above-mentioned material like the example 1. Since initial concentration was as low as 1.05 and electrification was poor when tested by ****(ing) like an example 2, the amount of electrifications on a sleeve had only 4 mC(s)/kg. Moreover, drum weld was also generated in per 100th sheet.

[0082]

[Effect of the Invention] Above, like explanation, according to this invention, there are not sleeve contamination, poor cleaning, and poor electrification, and the 1 component toner for electrostatic-charge image development which can continue maintaining a good picture can be obtained from the property excellent in endurance.

[Translation done.]